QUALITY SYSTEMS
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5.0 QUALITY SYSTEMS

INTRODUCTION

Quality Systems include all quality assurance (QA) policies and quality control (QC) procedures, which shall be delineated in a QA Plan to help ensure and document the quality of the analytical data. Laboratories seeking accreditation under NELAP must assure implementation of all QA policies and the essential applicable QC procedures specified in this chapter. The QA policies, which establish essential QC procedures, are applicable to environmental laboratories regardless of size and complexity. The laboratory shall meet any additional or more stringent requirements as specified by the analytical methods, specific programs or Agencies.

It is the intent of this section to provide sufficient detail concerning QA and QC requirements so that all accrediting authorities evaluate laboratories consistently and uniformly.

Section 5 is organized based upon ISO Guide 25, 1990. In some cases the names of major headings are slightly different than Guide 25, however, the content of such sections meets the intent of the ISO Guide 25 document. Where deemed necessary, specific areas within this section may contain more information than required in ISO Guide 25.

All items identified in this discussion shall be available for on-site inspection or data audit.

5.1 SCOPE

5.1.1 These standards specify the essential activities, records and procedures that a laboratory must implement to be considered for accreditation under NELAP.

5.1.2 If more stringent standards or requirements are specified by the test method or by regulation, the laboratory shall demonstrate that such requirements are met.

5.2 REFERENCES

See Appendix A

5.3 DEFINITIONS

See Appendix B

5.4 ENVIRONMENTAL LABORATORY ORGANIZATION AND MANAGEMENT

5.4.1 Legal Definition of Laboratory

The laboratory shall meet the criteria outlined in 1.6.1.1.

5.4.2 Organization

The laboratory shall:

a) Have available a clear description of the lines of responsibility in the laboratory and shall be proportioned such that adequate supervision is ensured. An organizational chart is recommended.

b) Have job descriptions shall be available for all positions.

c) Have a technical director (however named) who has the technical responsibility for the operation of the laboratory. The director shall certify that personnel with appropriate educational and/or technical background perform all analysis for which the laboratory is certified. Such certification shall be documented.

   The technical director shall:

   i. have a bachelor's degree in biological, chemical, or physical sciences from an accredited institution of higher education, plus three years experience in a certified environmental laboratory or its equivalent, as determined by the accrediting authority, with at least one of the three years in a supervisory capacity;

   or

   ii. in the case of a full-time employee of a domestic wastewater treatment facility whose laboratory is devoted exclusively to the testing of environmental samples taken from the facility, hold a valid treatment-plant operator's certificate appropriate to the nature and size of the facility. The analyses performed by such a laboratory shall be limited to the determination of percent moisture, biochemical oxygen demand, total

solids, suspended solids, volatile suspended solids, settleable solids, pH, temperature, dissolved oxygen, alkalinity, acidity, total chlorine residual and fecal coliform organisms;

or

ii. in the case of a full-time employee of an industrial wastewater treatment facility whose laboratory is devoted exclusively to the testing of environmental samples taken from the facility, have a minimum of one year experience in environmental analyses under acceptable supervision. The analyses performed by such a laboratory shall be limited to the determination of percent moisture, biochemical oxygen demand, total solids, suspended solids, volatile suspended solids, settleable solids, pH, temperature, dissolved oxygen, alkalinity, acidity, total chlorine residual and fecal coliform organisms.

d) A quality assurance officer (and/or his designee) who shall:

i. serve as the focal point for QA/QC and be responsible for the oversight and/or review of quality control data;

ii. have functions independent from laboratory operations for which they have Quality Assurance oversight;

iii. be able to objectively evaluate data and perform assessments without outside (e.g., managerial) influence;

iv. have documented training and/or experience in QA/QC procedures and be knowledgeable in the quality system as defined under NELAP;

v. have a general knowledge of the analytical methods for which data review is performed; and

vi. conduct internal audits on the entire operation annually.

e) Have contingency plans in the event that either the technical director or quality assurance officer is absent.

5.4.3 Performance Evaluation Samples

For purposes of qualifying for accreditation, each laboratory shall participate in a performance evaluation program as outlined in Chapter 2.0.

5.5 QUALITY SYSTEM - ESTABLISHMENT, AUDITS, ESSENTIAL QUALITY CONTROLS AND DATA VERIFICATION

5.5.1 Establishment

The laboratory shall establish and maintain a quality assurance system appropriate to the type, range and volume of testing activities it undertakes:

a) The elements of this system shall be documented.

b) The quality assurance documentation shall be available for use by the laboratory personnel.

c) The laboratory shall define and document its policies and objectives for, and its commitment to good laboratory practice and quality of testing services.

d) The laboratory management shall ensure that these policies and objectives are documented in a quality assurance system manual and communicated to, understood, and implemented by all laboratory personnel concerned.

e) The quality assurance system manual shall be maintained current under the responsibility of the quality assurance officer.

5.5.2 Quality Assurance Manual

The quality assurance manual, and its related quality assurance documentation, shall state the laboratory's policies and operational procedures established in order to meet the requirements of this Document.

The quality assurance manual and related quality assurance documentation shall also contain:

a) Identification of laboratory submitting quality assurance manual listing: document title; laboratory's full name and address; name, address (if different from above), and telephone number of individual responsible for the laboratory; quality assurance officer; identification of all major organizational units which are to be covered by this quality assurance manual; signed concurrences, with their appropriate identification, of all responsible parties including the QA

officer, laboratory director, laboratory owner, State/EPA regulatory officials;

Editorial Note: Laboratory director and laboratory owner are not defined. The defined term is manager.

b) Introduction giving a brief background, purpose, and scope of the quality assurance system;

c) a quality assurance policy statement, including objectives and commitments, by top management;

d) the organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts;

e) the relation between management, technical operations, support services and the quality assurance system;

f) job descriptions of key staff and reference to the job descriptions of other staff;

g) arrangements for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;

h) reference to the procedures for calibration, verification and maintenance of equipment;

i) reference to the facilities, services and major equipment as well as reference measurement standards used;

j) the laboratory's scope of analytical tests;

k) reference to the calibration, verification and/or test procedures used;

l) procedures for handling submitted samples;

m) the laboratory's procedures for achieving traceability of measurement;

n) procedures for control and maintenance of documentation including: laboratory notebooks; instrument logbooks;

standards logbooks; data reduction, validation storage and reporting;

o) reference to verification practices including interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes;

p) procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures form documented policies and procedures occur;

q) the laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications;

r) procedures for dealing with complaints;

s) procedures for protecting confidentiality and proprietary rights;

t) training;

u) safety;

v) laboratory waste; and

w) procedures for audit and review.

5.5.3 Audits

5.5.3.1 Managerial Review

The quality assurance system adopted to satisfy the requirements of this Document shall be reviewed at least once a year by the management to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements.

5.5.3.2 Internal Audits

The laboratory shall arrange for annual audits of its activities to verify that its operations continue to comply with the requirements of the quality assurance system. Such audits shall be carried out by the quality assurance officer and/or designee(s) who are trained and qualified and who are, whenever possible, independent of the activity to be audited. Where the audit findings cast doubt on the

correctness or validity of the laboratory's calibrations or test results, the laboratory shall take immediate corrective action and shall immediately notify, in writing, any client whose work may have been affected.

5.5.3.3 Audit Review

All audit and review findings and any corrective actions that arise from them shall be documented. The Quality Assurance Officer shall ensure that these actions are discharged within the agreed timescale.

5.5.3.4 Performance Audits

In addition to periodic audits, the laboratory shall ensure the quality of results provided clients by implementing checks. These checks shall be reviewed and shall include, as appropriate, but not be limited to:

a) internal quality control schemes using whenever possible statistical techniques (see 5.5.4 below);

b) participation in proficiency testing or other interlaboratory comparisons;

c) regular use of certified reference materials and/or in-house quality control using secondary reference materials;

d) replicate testing using the same or different methods;

e) re-testing of retained items;

f) correlation of results for different characteristics of an item.

5.5.3.5 Corrective Actions

a) In addition to providing acceptance criteria and specific protocols for corrective actions in the Method Standard Operating Procedures (see 5.10.2.2), The laboratory shall implement general procedures to be followed to determine when quality control data is out of control:

    i. Identify the individual(s) responsible for assessing each QC data type;

ii. Identify the individual(s) responsible for initiating and/or recommending corrective actions;

iii. Define how the analyst should treat a data set if the associated QC measurements are unacceptable;

iv. Specify how out-of control situations and subsequent corrective actions are to be documented; and

v. Specify procedures for management (including the QA officer) to review corrective action reports.

5.5.4 Essential Quality Control Procedures

These general quality control principles shall apply, where applicable, to all testing laboratories. The manner in which they are implemented is dependent on the types of tests performed by the laboratory (i.e., chemical, microbiological, radiological). The standards for any given test type shall assure that these applicable principle concepts are addressed:

a) All laboratories shall have protocols in place to monitor the following quality controls:

i. Adequate positive and negative controls to monitor tests such as blanks, spikes, reference toxicants, zero blanks;

ii. Adequate tests to define the variability and/or reproducibility of the laboratory results and/or samples such as duplicates;

iii. Measures to assure the accuracy of the test data including sufficient calibration and/or continuing calibrations, use of certified reference materials, performance evaluation samples, or other measures;

iv. Determinations of test sensitivity, such as method detection limits or mortality, and range of applicability such as linearity;

v. Selection of appropriate formulae to reduce raw data to final results such as linear regression, internal standards, or statistical packages;

vi. Selection and use of reagents and standards of appropriate quality;

vii. Measures to assure the selectivity of the test for its intended purpose; and

viii. Measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the method such as temperature, humidity, light, or specific instrument conditions.

b) All quality control measures shall be assessed and evaluated on an on-going basis, and quality control acceptance limits shall be used to determine the validity of the data. The acceptance/rejection criteria shall be updated at a frequency established by the method or by NELAP standards.

c) The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exists.

d) The method specified and/or method-recommended quality control protocols shall be followed. The essential standards outlined in the following sections shall be used if no protocols are written into the method or if the method protocols are less stringent.

5.5.4.1 Chemical Testing

5.5.4.1.1 Positive and Negative Controls

a) Negative Controls

i. Method Blanks - A minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation.

b) Positive Controls

i. Matrix Spikes (MS) - Shall be performed at a frequency of one per batch of 20 or less samples per matrix type per sample extraction or preparation except for analytes for which standards are not available such as BOD, CBOD, TSS, TDS, TVS, Total Solids, oil and grease, pH, color, odor, temperature, dissolved or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client of the sample.

ii. Laboratory Control Sample - (QC Check Samples) Shall be analyzed at a minimum of 1 per batch of 20 or less samples per matrix type per sample except for analytes for standards are not available such as BOD, CBOD, TSS, TDS, TVS, Total Solids, oil and grease, pH, color, odor, temperature, dissolved or turbidity. The results of these samples shall be used to determined batch acceptance.

iii. Surrogates - Surrogate compounds must be added to all samples, standards, and blanks, whenever possible, for all organic chromatography methods.

5.5.4.1.2 Laboratory Variability/Reproducibility

Matrix Spike Duplicates (MSDs) or Laboratory Duplicates (SD) -Shall be analyzed at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation. The laboratory shall document their procedure to select the use of appropriate type of duplicate.

5.5.4.1.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Initial Demonstration of Analytical Capability (Section 5.10.2.1) shall be performed initially and with a significant change, such as a new analyst, instrument or technique.

b) Calibration - Calibration protocols specified in Section 5.10.1 shall be followed.

c) Performance Evaluation Samples - The results of such analyses (5.4.3 or 5.5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

5.5.4.1.4 Sensitivity

a) Method detection Limits - Method detection limits shall be determined by 40 CFR Part 136, Appendix B unless specified by a method or program. The detection limit shall be determined for the compounds of interest in each method in laboratory pure water or the matrix of interest and shall be verified annually. All procedures used must be documented.

5.5.4.1.5 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

5.5.4.1.6 Quality of Standards and Reagents

a) The source of standards shall comply with 5.9.1.

b) Reagent Quality, Water Quality and Checks:

i. Reagents - In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than that specified by the method shall not be used. The labels on the container should be checked to verify that the purity of the reagents meets the needs of the particular method.

ii. Water - The quality of water sources shall be monitored and documented.

5.5.4.1.7 Selectivity

a) A confirmation shall be performed to verify the compound identification when positive results are detected on a new matrix, new analyte, or when historical test knowledge of the sample is not available. Such confirmations shall be performed on organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical method. Confirmation is required unless stipulated in writing by the client.

5.5.4.1.8 Constant and Consistent Test Conditions

a) The laboratory shall assure that the test instruments consistently operate within the specifications of the test methods and equipment manufacturer.

b) Glassware Cleaning - Glassware shall be cleaned to meet the sensitivity of method.

Any cleaning and storage procedures that are not specified by the method shall be documented in laboratory records and SOPs.

5.5.4.2 Whole Effluent Toxicity

5.5.4.2.1 Positive and Negative Controls

a) Positive Control - Reference Toxicants - Reference toxicant tests indicate the sensitivity of the test organisms being used and demonstrate a laboratory's ability to obtain consistent results with the method.

i. The laboratory must demonstrate its ability to obtain consistent, results with reference toxicants before it performs toxicity tests with effluents for permit compliance purposes.

   a. The intralaboratory precision, expressed as percent coefficient of variation (CV%), shall be determined by performing five or more tests with different batches of test organisms, using the same reference toxicant, at the same concentrations under the same test conditions (i.e., the same test duration, type of dilution water, age of test organisms, feeding), and the same statistical analysis.

   b. Intra-laboratory precision on an ongoing basis must be determined through the use of reference toxicant tests and plotted in quality control charts. As specified in the test methods, the control charts shall be plotted as point estimate values, such as EC25 for chronic tests and LC 50 for acute tests, over time within a laboratory.

ii. Frequency:

   a. Reference toxicant tests shall performed concurrently with the effluent toxicity tests when using test organisms from outside the test laboratory. If organisms are obtained from an outside source, a single reference toxicant test is acceptable for comparison with multiple effluent tests if:
      1) all tests are conducted concurrently;
      2) test conditions are the same for all tests; and
      3) all organisms are from a single group spawned, collected or released from the same population at the same time.

   b. Reference toxicant tests shall be performed at least once a month if using test organisms cultured within the testing laboratory.

iii. The USEPA test methods for EPA/600/4-91-002, EPA/600/4-91-003 and EPA/600/4-90-027F do not currently specify a particular reference toxicant and dilution series,

however, if the state or permitting authority identifies a reference toxicant or dilution series for a particular test, the laboratory shall follow the specified requirements.

iv. Test Acceptability Criteria (TAC) - The test acceptability criteria such as the water flea test, requires 80% or greater survival and an average 15 young per female in the controls) as specified in the test method must be achieved for both the reference toxicant and effluent test. The criteria shall be calculated and shall meet the method specified requirements for performing toxicity:

a. The control population of *Ceriodaphnia* shall contain no more than 20% males.

b. An individual test may be conditionally acceptable if temperature, dissolved oxygen, pH and other specified conditions fall outside specifications, depending on the degree of the departure and the objectives of the tests (see test conditions and test acceptability criteria specified for each test method). The acceptability of the test shall depend on the experience and professional judgment of the technical employee and the permitting authority.

b) Negative Control - Control, Brine Control or Dilution Water - The standards for the use, type and frequency of testing are specified by the methods and by permit and shall be followed.

5.5.4.2.2 Variability and/or Reproducibility

Intra-laboratory precision shall be determined on an ongoing basis through the use of further reference toxicant tests and related control charts as described in item 5.5.4.2.1 a) above.

5.5.4.2.3 Accuracy

This principle is not applicable to Whole Effluent Toxicity.

5.5.4.2.4 Test Sensitivity

a) Test sensitivity (or test power) of the tests will depend in part on the number of replicates per concentration, the significance level selected (0.05), and the type of statistical analysis. If the variability remains constant, the sensitivity of the test will increase as the number of replicates is increased. Test sensitivity is the minimum significant difference (MSD) between the control and test concentration that is statistically significant. The MSD shall be calculated and reported with the test results where the Dunnett's procedure is used.

b) In order to have sufficient replicates to perform a reliable MSD, such tests will have a minimum of four replicates per treatment.

5.5.4.2.5 Selection of Appropriate Statistical Analysis Methods

a) The methods of data analysis and endpoints will be specified by language in the permit or, if not present in the permit, by the EPA methods manuals for Whole Effluent Toxicity.

b) Dose Response Curves - When required, the data shall be plotted in the form of a curve relating the dose of the chemical to cumulative percentage of test organisms demonstrating a response such as death.

5.5.4.2.6 Selection and Use of Reagents and Standards

a) The grade of all reagents used in WET are specified in the method except the reference standard. All reference standards shall be prepared from chemicals which are analytical reagent grade or better.

b) All standards and reagents associated with chemical measurements, such as dissolved oxygen, pH or specific conductance, shall comply with the standards outlined in 5.5.4.1 above.

5.5.4.2.7 Selectivity

This principle is not applicable. The selectivity of the test is specified by permit.

5.5.4.2.8 Constant and Consistent Test Conditions

a) If closed refrigerator-sized incubators are used, culturing and testing of organisms shall be separated to avoid loss of cultures due to cross-contamination.

b) The laboratory or a contracted outside expert shall positively identify test organisms to species on an annual basis. The taxonomic reference (citation and page(s))and the names(s) of the taxonomic expert(s) must be kept on file at the laboratory.

c) Instruments used for routine measurements of chemical and physical parameters such as pH, DO, temperature, conductivity, salinity, alkalinity, hardness, chlorine, and weight shall be calibrated, standardized and documented prior to use each day as outlined in the Section 5.5.4.1.

d) Test temperature shall be maintained as specified in the methods manuals. The average daily temperature of the test solutions must be maintained within 1°C of the selected test temperature, for the duration of the test. The minimum frequency of measurement shall be once per 24 hour period.

e) Water used for culturing and testing shall be analyzed for toxic metals and organics annually or whenever the minimum acceptability criteria for control survival, growth or reproduction is not met and no other cause, such as contaminated glassware or poor stock, can be identified.

   i. The concentration of the metals, Al, As, Cr, Co, Cu, Fe, Pb, Ni, Zn, expressed as total metal, shall not exceed 1 ug/l each.

   ii. Cd, Hg, and Ag, expressed as total metal shall not exceed 100 ng/l each.

   iii. Total organochlorine pesticides plus PCBs shall be less than 50 ng/l. Individual pesticide concentrations shall not exceed EPA's Ambient Water Quality chronic criteria where available.

f. New batches of food used for culturing and testing shall be analyzed for toxic organics and metals. If food combinations or recipes are used, analyses shall be performed on the final product upon the use of new lot of any ingredient. If the concentration of total organic chlorine exceeds 0.15 ug/g wet

weight, or the total concentration of organochlorine pesticides plus PCBs exceeds 0.30 ug/g wet weight, or toxic metals exceeds 20 ug/g wet weight, the food must not be used.

g) Test chamber size and test solution volume shall be as specified in the methods manuals.

h) Test organisms shall be fed the quantity and type food specified in the methods manuals. They shall also be fed at the intervals specified in the methods.

i) Light intensity shall be maintained as specified in the methods manuals. Measurements shall be made and recorded on a yearly basis. Photoperiod shall be maintained as specified in the methods.

j) At a minimum, during chronic testing DO and pH shall be measured in at least one replicate of each concentration.

k) All cultures used for testing shall be maintained as specified in the methods manuals.

l) Age and the age range of the test organisms must be as specified in the manuals.

m) The maximum holding time (lapsed time from sample collection to first use in a test) shall not exceed 36 hours without the permission of the permitting authority.

n) All samples shall be chilled during or immediately after collection. They shall be maintained at and the arrival temperature shall be 0.1 to 6°C. Samples that are hand delivered to the laboratory immediately after collection (i.e., within 1 hour) may not meet the laboratory temperature acceptance criteria. In these cases, the laboratory may accept the samples if there is evidence that the chilling process has begun such as arrival on ice.

o) Organisms obtained from an outside source must be from the same batch.

5.5.4.3 Microbiology

Microbiological analysis, recovery or testing involves he culture medium, the sample and the microbial species being isolated or tested.

5.5.4.3.1 Positive and Negative Controls

a) Negative Controls
   i. Blanks (Sterility checks) - The uninoculated controls specified by each method shall be run in the manner and frequency specified.

b) Positive Controls - At least one pure culture of a known positive reaction shall be tested regularly month for each test, or more appropriately, included in the test procedure.

5.5.4.3.2 Laboratory Variability/Reproducibility

a) Duplicates - At least 5% of the positive samples shall be duplicated. In laboratories with more than one analyst, each shall make parallel analyses on at least one positive sample per month.

b) Participation in internal or inter-laboratory collaborative studies must be done when possible.

5.5.4.3.3 Accuracy

This principle is not applicable to microbiology

5.5.4.3.4 Sensitivity

All growth and recovery media must be checked to assure that the target organisms respond in an acceptable and predictable manner (see 5.5.4.3.1.b).

5.5.4.3.5 Data Reduction

a) The calculations, data reduction and statistical interpretations specified by each method shall be followed.

b) If the method specifies colony counts, such as membrane filter or colony counting, then the ability of individual analysts to identify and count colonies shall be verified at least once per month, by having two or more analysts count colonies from the same plate.

c) Use of Automated Plate Counters *Mary to provide standards*

5.5.4.3.6 Quality of Reagents and Media

a) Laboratory Pure (Reagent) Water Requirements

i. The quality of water used by a laboratory shall be controlled with regular suitability tests for bactericidal properties of distilled or other laboratory pure water.

ii. Laboratory pure water shall be analyzed regularly (per method requirements) for pH, chlorine residual, microbiologic standard plate count and specific conductance.

iii. Laboratory pure water must be analyzed annually for trace metals.

b) The laboratory media shall be expiration dated and each lot regularly tested for growth promotion and lack of inhibitory activity.

5.5.4.3.7 Selectivity and Specificity

a) When the MPN test is performed on environmental samples, at least 10% of the positive samples shall be confirmed using the specified methods and requirements.

b) When Membrane Filter tests are performed, the results shall be verified with specific requirements utilizing positive and negative controls.

c) Microorganisms from an appropriately certified source (e.g., American Type Culture Collection) shall be used to confirm the morphological and biochemical responses to test media. The laboratory shall have a procedures to verify such cultures and records of the certified sources and verification shall be maintained.

d) Visual identification of organisms *Mary to Provide general standards*

Methods for handling air samples and MPA need to be addressed at a future date when techniques have been formalized

5.5.4.3.8 Constant and Consistent Test Conditions

All test conditions, such as incubator and refrigerator temperatures, water bath temperatures and media preparation, shall follow method specified protocols and shall use serviced and calibrated equipment.

5.5.4.4 Radioanalysis

Analysis of Quality Control Samples - A radioanalytical laboratory shall analyze both intralaboratory and interlaboratory quality control samples on a routine basis as described in the laboratory's Quality Assurance Plan. Quality control samples shall account for at least l0 percent of the total analytical load and shall not be limited to one type or category. Where possible, the laboratory shall employ the use of blind and double-blind quality control samples.

5.5.4.4.1 Positive and Negative Controls

a) Negative Controls - Reagent Blank - The laboratory shall analyze reagent blanks so as to provide a means of evaluating and quantifying potential contamination resulting from the samples' passage through the analytical process. The volume or weight of the blank shall be approximately equal to the volume or weight of samples routinely analyzed, and the blanks shall be carried through the entire analytical process.

b) Positive Control - Matrix Spike - The laboratory shall analyze matrix spike samples to evaluate the effect of the sample matrix upon the analytical methodology. The activity level of the matrix spike sample shall be comparable to the activity levels of samples routinely analyzed. The laboratory shall prepare an analyze matrix spike samples for each type of matrix analyzed.

5.5.4.4.2 Laboratory Variability/Reproducibility

Replicate - The laboratory shall perform replicate analyses to evaluate the precision of an analysis. The replicate analyses shall include replicates of actual samples, replicates of

matrix spike samples and replicates of traceable reference material. The size of replicate samples shall be approximately equal to the size of samples routinely analyzed.

5.5.4.4.3 Method Evaluation

a) Traceable Reference Material - The laboratory shall analyze traceable reference materials to evaluate the accuracy and precision of an analytical methodology or an analyst. Traceable Reference Material, as defined by ANSI N42.2 *Measurement Quality Assurance For Radioassay Laboratories* is a NIST prepared standard reference material (SRM) or a sample of known concentration prepared from a NIST traceable reference material (derived standard material).

b) Calibration of Radiation Measurement Systems - Performance assessments shall, at a minimum, utilize the following practices: (l) baselining each measurement system's response characteristics, (2) stability check with control chart plot, and (3) background check with control chart plot. The frequency of the performance assessments must be sufficient to promptly identify and correct invalid results and must also consider radiation measurement system (RMS). For a RMS which is used to identify radionuclides, the stability check shall also include geometry-specific energy-calibration sources. Background count rate checks shall be performed to ensure that the RMS is uncontaminated and that the observed count rate is consistent with a priori sensitivity assumptions. Warning and out-of-control limits and the exceedance investigation protocol shall be specified for each control chart maintained.

   Radiation instrumentation energy calibration (channel number of the multichannel analyzer versus the radiation energy),full-energy peak efficiency, radionuclide activity (concentration) precision and test method precision and bias should be checked periodically (typically monthly to yearly) with appropriate standard sources. Additional checks should be carried out whenever significant changes in the analytical instrumentation is detected.

5.5.4.4.4 Sensitivity

5.5.4.4.5 Data Reduction - Procedures utilized in the computation of final concentrations of radioactive materials shall include an independently derived verification of results.

5.5.4.4.6 Quality of Standards and Reagents

a) Radionuclide Reference Standards - A radiochemistry laboratory must have an operational internal quality control program that ensures that all radiation detection instruments are calibrated and functioning. The quality control program must have a radionuclide standards traceability program. Reference standards that are used in a radioanalytical laboratory must be obtained from either the National Institute of Standards and Technology (NIST) or suppliers who participate in supplying NIST standards or NIST traceable radionuclides. NIST standards must be accompanied with a certificate of calibration which describes the standard's (l) principle radionuclide, mass or volume, and chemical composition; (2) reference time and date; (3) measurement result (activity of principal and possible daughter radionuclides per gram of solution; (4) measurement method; (5) a statement of purity (list of known or suspected radionuclide impurities, their activities, and how they were measured); (6) decay information (statement of the assumed half-life and other decay information); and (7) and estimate of errors (includes errors from the measurements themselves and those created by the decay assumptions). In all radionuclide measurements, the volumes, shapes, and physical and chemical characteristics of the standards and their containers must be as identical as practicable for the most accurate results.

5.5.4.1.7 Selectivity

5.5.4.1.8 Constant and Consistent Test Conditions

5.5.4.5 Air Testing

Analyses for Air Toxics shall follow the essential quality controls for chemistry outlined in Section 5.5.4.1.

5.5.5 Data Verification - *Section needs to be added*

5.6 PERSONNEL

5.6.1 General requirements for laboratory staff

The testing laboratory shall have sufficient supervisory and other personnel, having the necessary education, training, technical knowledge and experience for their assigned functions.

5.6.2 Laboratory Staff Responsibilities and Credentials

Laboratory management shall be responsible for:

a) All analytical and operational activities of the laboratory, including those of any auxiliary or mobile laboratory facilities;

b) Supervision of all personnel employed by the laboratory, including those assigned to work in any auxiliary or mobile laboratory facilities, and those persons designated as principle analysts;

c) Assuring that all sample acceptance criteria (Section 5.9) are met and that samples are logged into the sample tracking system and properly labeled and stored; and

d) The production and quality of all data reported by the laboratory, including any auxiliary or mobile laboratory facilities.

e) Assuring that all laboratory staff have demonstrated initial and ongoing proficiency in the activities for which they are responsible. Such demonstration shall be documented.

Each analyst and other members of the staff shall be responsible for complying with all QA requirements. Each laboratory position must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular function and a general knowledge of laboratory operations, analytical methods, quality assurance/quality control procedures and records management.

5.6.3 Records on relevant qualifications, skills and experience of technical personnel shall be maintained.

5.7 PHYSICAL FACILITIES

5.7.1 Environment

Laboratory facilities, including fixed and mobile, shall be maintained to permit the production of analytical data of needed quality. In addition to housekeeping that must be performed to assure that contamination is unlikely, the following elements shall be considered:

- temperature;
- humidity;
- electrical power;
- vibration;
- electromagnetic fields;
- dust;
- direct sunlight;
- ventilation (exhaust hoods, air exchangers, etc.); and
- lighting.

In instances where environmental controls are specified by method, such as temperature, lighting, or humidity, the laboratory shall meet and document the method-specified criteria.

5.7.2 Work Area

Work spaces to ensure an unencumbered work area must be available. These include:

- controlled access to the laboratory;
- separation of incompatible analyses including culture areas;
- sample receipt area;
- sample storage area;
- chemical and waste storage area(s); and
- data handling and storage area(s).

5.8 EQUIPMENT AND REFERENCE MATERIALS

5.8.1 A laboratory must have access to all equipment and reference materials specified by the analytical procedures for which accreditation is sought.

5.8.2 The laboratory shall establish and follow a preventative maintenance program for all equipment.

5.8.3 All maintenance activities, both routine and nonroutine, shall be documented. The following records shall be maintained for each piece of equipment:

- Name of item;
- Manufacturer's name, type identification and serial number;
- Date received and placed in service;
- Current physical location;
- Maintenance log; and
- Calibration information, if appropriate.

5.9 MEASUREMENT TRACEABILITY AND CALIBRATION

All measurement or testing equipment which affect the accuracy or validity of the result (including balances, thermometers and reference standards) shall be calibrated and verified before use and on a continuing basis.

Issue: Should specific acceptance criteria be established for balances, thermometers and reference standards?

The laboratory shall have procedures in place to calibrate and verify all measuring or test equipment.

5.9.1 Traceability of Calibration

5.9.1.1 Where possible, such calibration or verification shall be traceable to national standards.

5.9.1.2 The laboratory shall maintain records of all such certifications including, where applicable, the measurement result, the associated uncertainty of the measurement, such as standard deviation or variance, and the statement of compliance with an identified specification.

5.9.1.3 If traceability to national standards is not possible, the laboratory shall demonstrate, by appropriate means such as proficiency testing, that the measurement equipment is properly calibrated.

5.9.2 Documentation and Labeling of Standards and Reagents

5.9.2.1 The laboratory shall retain records, such as manufacturer's statement of purity, of the origin, purity and traceability of all standards and reagents (including balance weights and thermometers). These records shall

include the date of receipt, and, if applicable, the date of opening and an expiration date.

5.9.2.2 Detailed records shall be maintained on reagent and standard preparation. These records shall indicate traceability to purchase stocks or neat compounds, and must include the date of preparation and preparer's initials.

5.9.2.3 Where calibrations do not include the generation of a calibration curve, such as thermometers, balances, or titrations, records shall indicate the calibration date and type (balance weight, thermometer serial number, primary standard concentration) of calibration standard that was used.

5.9.2.4 All prepared reagents and standards shall be clearly identified with preparation date, concentration(s) and preparer's initials.

5.9.2.5 All calibration curves shall be dated and labeled with method, analyte and standard concentrations and instrument responses.

5.9.2.6 The axes of the calibration curve shall be labeled. For electronic data processing systems, that automatically compute the calibration curve, the equation for the curve and the correlation coefficient must be recorded. The equation for the line and the correlation coefficient shall also be recorded when the calibration curve is prepared manually.

5.9.2.7 A criteria for calibration acceptance such as an acceptable correlation coefficient shall be established and documented.

5.10 SAMPLE CALIBRATION AND TEST METHODS

5.10.1 Calibration

5.10.1.1 Initial Calibrations

When available, all initial calibrations shall be verified with standards obtained from a second or different source. These

verification standards shall be analyzed with each initial calibration.

Calibration curves shall be prepared as specified in the method.

If a method does not provide guidance in the preparation of a calibration curve, the following protocols shall be followed:

a) At least a blank and three (3) standards that lie within the linear portion of the curve shall be used.

b) The lowest standard shall be at a concentration approaching the lowest quantifiable level for the method (approximately four to eight times the calculated method detection limit).

c) Additional standards shall be required for non-linear calibration curves.

d) In all cases, the sample results must be closely bracketed by calibration standards.

e) A new curve shall be run if two successive runs of one continuing calibration check is outside acceptable limits.

5.10.1.2 Continuing Calibration Verification

This section remains unchanged pending further discussions

When an initial calibration curve is not run on the day of analysis, the integrity of the initial calibration curve shall be verified on each day of use (or 24 hour period) by initially analyzing a blank and a standard at a concentration equal to or near the lowest calibration standard (the lowest calibration standard shall be in the range of 4 to 8 times the calculated method detection limit).

Additional standards shall be analyzed after the initial calibration curve or the integrity of the initial calibration curve (see previous paragraph) has been accepted.

a) These standards shall be analyzed at a frequency of 5% or every 12 hours whichever is more frequent and may be standards used in the original calibration curve or standards from another source.

b) The concentration of these standards shall be determined by the anticipated or known concentration of the samples and/or method specified levels. To the extent possible, the samples in each interval (i.e. every 20 samples or every 12 hours) should be bracketed with standard concentrations closely representing the lower and upper range of reported sample concentrations. If this is not possible, the standard calibration checks should vary in concentration throughout the range of the data being acquired.

When not specified by the analytical method, these calibration verification standards shall be within 15% of the true value.

5.10.2 Test Methods and Standard Operating Procedures

When the use of approved methods for a specific sample matrix is required, only those methods shall be used. In addition, where performance-based methods or non-legally mandated methods are permitted, the relevant start-up and ongoing validation procedures, and calibrations as specified in 5.10.2.1 must be followed and documented.

The criteria listed in 5.10.2 must be met for all methods and SOPs.

5.10.2.1 Method Validation/Initial Demonstration of Method Performance

Prior to acceptance and institution of any method, satisfactory initial demonstration of method performance, in conformance with the relevant EPA guidelines, is required. In the absence of method-specified requirements, this demonstration shall follow the outlined protocols of Paragraph 8.1.1 and Section 8.2 in the methods published in 40 CFR Part 136, Appendix A. Thereafter, continuing demonstration of method performance, in conformance with the relevant EPA guidelines, is required. In both cases, the appropriate standard performance checklist (see Appendix C) must be completed and retained by the laboratory to be made available upon request. All associated supporting data necessary to reproduce the analytical results summarized in the checklists must be retained by the laboratory. Initial demonstration of method performance must be completed each time there is a change in equipment, personnel or procedure.

5.10.2.2 Laboratory Method Manual(s)

a) The laboratory shall have and maintain an in-house methods manual(s) which, along with any associated reference works, shall be available to the bench analyst.

b) For each analyte or test to be certified, a method or methods to be used by the laboratory shall be described in a methods manual which may cross reference other laboratory SOPs. The method description shall include where applicable:

- identification of the test method and where applicable, the analyte name with qualifier (the qualifier is a word, phrase or number that better identifies the method; e.g., "Iron, Total", or "Chloride, Automated Ferricyanide", or "Our Lab. Method SOP No. 101");
- applicable matrix or matrices;
- method detection limit;
- scope and application;
- summary of the method;
- definitions;
- interferences;
- safety;
- equipment and supplies;
- reagents and standards;
- sample collection, preservation, shipment and storage;
- quality control;
- calibration and standardization;
- procedure;
- calculations;
- method performance;
- pollution prevention;
- data assessment and acceptance criteria for quality control measures;
- corrective actions for out-of-control data;
- contingencies for handling out-of-control or unacceptable data;
- waste management;
- references; and
- any tables, diagrams, flowcharts and validation data

c) In cases where minor modifications to accepted methods have been made such as change in type of column or change in operating conditions, or where the referenced method is

ambiguous or provides insufficient detail such as reagent purity or reagent concentration, these changes or clarifications shall be documented as an appendix to the referenced method.

5.10.2.3 Standard Operating Procedures (SOPs)

In addition to the method manuals (5.10.2.2), organizations shall maintain standard operating procedures that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions or handling customer complaints.

a) These documents may be equipment manuals provided by the manufacturer, or internally written documents.

b) The SOPs shall also include a list of analytical methods that are used by the laboratory. This list shall be indexed according to NELAC accreditation categories.

c) Copies of all SOPs shall be accessible to the workplace.

5.10.3 Computers and Electronic Data Related Requirements

Environmental laboratories are increasingly employing microprocessors and computers in the measurement and documentation processes. The Agency has developed Good Automated Laboratory Practices (GALPs), 199? And 199?, EPA #??????,# ??????. These documents were developed over time as computer and electronic data experts developed a listing of procedures essential to help assure data integrity, reliability and defensibility. The GALPs have been internationally accepted and are consistent with the electronic data requirements necessary for environmental laboratories.

These GALP requirements have been adopted by NELAC without any technical changes but with an important expansion of scope. The Agency's GALPs limit the scope of the requirements to laboratories employing formal Laboratory Information Management System. As environmental laboratories vary widely in degree of electronic automation from those with microprocessor driven pH meters to facilities with extensive computerization/automation, to still others with advanced LIMS systems to manage data and generate reports, the essential provisions delineated in the current version (???????)of the EPA Office of information Managements's "Good Automated Laboratory Practices (Practices and Guidelines for Ensuring Data Integrity in Automated Laboratory Operations with

Implementation Guidance)" shall be followed in the laboratory's data measurement and documentation processes.

These GALP practices apply to all laboratories employing microprocessors and computers and are not limited to facilities with LIM Systems.

5.11 SAMPLE HANDLING SAMPLE ACCEPTANCE POLICY AND SAMPLE RECEIPT

Regardless of the laboratory's level of control over sampling activities, the following are essential to ensure sample integrity and valid data.

5.11.1 Sample Acceptance Policy

The laboratory shall have a written sample acceptance policy that clearly outlines the circumstances under which samples will be accepted. Data from any samples which do not meet the following criteria must be flagged in an unambiguous manner clearly defining the nature and substance of the variation. This document shall be circulated to sample collecting personnel and shall include, but is not limited to, the following areas of concern:

a) Proper, full, and complete documentation, which shall include sample identification, the location, date and time of collection, collector's name, preservative added, sample type and any special remarks concerning the sample;

b) Proper sample labeling to include unique identification and a labeling system for the samples with requirements concerning the durability of the labels (water resistant) and the use of indelible ink;

c) Evidence of proper preservation and use of appropriate sample containers.

d) Adherence to specified holding times; and

e) Adequate sample volume. Sufficient sample volume must be available to perform the necessary analysis.

5.11.2 Sample Receipt Protocols

5.11.2.1 Samples shall be checked upon receipt for all items specified in 5.11.1 above.

a) All samples which require thermal preservation shall be considered acceptable if the arrival temperature is within +/- 2°C of the required temperature. For samples with a specified temperature of 4°C, samples with a temperature of 0.1 to 6°C shall be acceptable. Samples that are hand delivered to the laboratory immediately after collection may not meet this criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice.

b) Chemical preservation such as appropriate pH) shall be checked upon receipt or prior to sample preparation/analyses.

5.11.2.2 The results of all checks shall be recorded.

5.11.2.3 If the sample does not meet the sample receipt acceptance criteria, the laboratory shall either:

a) Retain all correspondence and/or official conversations concerning the final disposition of rejected samples;

or

b) Fully document any decision to proceed with the analysis of compromised samples:
- The condition of these samples shall, at a minimum, be noted on the chain of custody or transmittal form and laboratory receipt documents.
- The analysis data shall be appropriately "qualified as estimated" on the final report.

5.11.2.4 The laboratory shall utilize a permanent, chronological log to document receipt of all sample containers. The following information must be recorded in the laboratory sequential log:
- Date of laboratory receipt of sample;
- Sample collection date;
- Unique laboratory ID code (see 5.11.4);
- Field ID code supplied by sample submitter;
- Requested analyses, including approved method number, if applicable;
- Signature or initials of data logger;
- Comments resulting from inspection for sample acceptance rejection; and
- Sampling kit code (if applicable).

5.11.2.5 All documentation, such as memos or transmittal forms, that is transmitted to the laboratory by the sample transmitter shall be retained.

5.11.2.6 If applicable, a complete chain of custody record (Section 5.12.4) shall be maintained.

5.11.3 Storage Conditions

5.11.3.1 Samples shall be stored according to the conditions specified by preservation protocols:

a) Samples which require thermal preservation shall be stored under refrigeration which is +/- 2° of the specified preservation temperature.

b) Samples for volatile organics shall be stored under refrigeration (see 5.11.3.1.a above) and separately all other samples or standards.

c) Samples shall be stored away from all standards, reagents, food and other potentially contaminating sources such as cleaning supplies or fuels.

5.11.3.2 Sample fractions, extracts, leachates and other sample preparation products shall be stored according to 5.11.3.1 above or according to specifications in the method.

5.11.3.3 Samples shall be stored in a secure area.

5.11.4 Sample Tracking

The laboratory shall design a system to unequivocally identify all samples, subsamples and subsequent extracts and/or digestates so that each aliquot is uniquely identified.

5.11.4.1 The laboratory shall assign a unique identification (ID) code to each sample container received in the laboratory. Multiple aliquots of a sample that have been received for different analytical tests, such as nutrients, metals, or VOCs, must be assigned a different ID code. The use of container shape, size or other physical characteristic, such as amber glass, or purple top, is not an acceptable means of identifying the sample.

5.11.4.2 This laboratory code shall maintain an unequivocal link with the unique field ID code assigned each container.

5.11.4.3 The laboratory ID code shall be placed on the sample container as a durable label.

5.11.4.4 The laboratory ID code shall be entered into the laboratory records (see 5.11.2.4) and shall be the link that associates the sample with related laboratory activities such as sample preparation or calibration.

5.11.4.5 In cases where the sample collector and analyst are the same individual or the laboratory preassigns numbers to sample containers, the laboratory ID code may be the same as the field ID code.

5.12 RECORD KEEPING

The laboratory shall implement protocols that will produce unequivocal, accurate records which document all laboratory activities associated with sample receipt, preparation, analysis, review and reporting.

There are two levels of record keeping: 1) sample custody or tracking and 2) legal or evidentiary chain of custody. All essential requirements for sample custody are outlined in Sections 5.12.1, 5.12.2 and 5.12.3. The basic requirements for legal chain of custody (if required or implemented) are specified in Section 5.12.4.

5.12.1 Record Keeping System and Design

Each organization shall design and maintain a record keeping system that is succinct, self-explanatory and efficient and allows historical reconstruction of all laboratory activities that produced the resultant sample analytical data. The history of the sample must be readily understood through the documentation. This shall include interlaboratory transfers of samples and/or extracts.

5.12.1.1 All information relating to the laboratory facilities equipment, analytical methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification shall be documented.

5.12.1.2 The record keeping system shall facilitate the retrieval of all working files and archived records for inspection and verification purposes.

5.12.1.3 All documentation entries shall be signed or initialed by responsible staff. The reason for the signature or initials shall be clearly indicated in the records (e.g., sampled by, prepared by, reviewed by, etc.).

5.12.1.4 All data generated during the conduct of a study, except those that are generated by automated data collection systems, shall be recorded directly, promptly and legibly in ink.

5.12.1.5 Entries in records shall not be obliterated by methods such as erasures, overwritten files or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction. These criteria also shall apply to electronically maintained records.

5.12.2 Records Management and Storage

5.12.2.1 All records of an organization that are pertinent to a specified project shall be retained for a minimum of five years unless otherwise designated for a longer period of time in another regulation. The records specified in 5.12.3 and 5.12.4 above shall be retained.

5.12.2.2 Records that are stored or generated by computers or personal computers (PCs) shall have hard copy and write-protected backup copies.

5.12.2.3 When a procedure or document, such as initial calibration records or SOPs, becomes obsolete or is replaced, the records shall clearly indicate the time period (or sample sets, if applicable) during which the procedure or document was in force.

5.12.2.4 Access to archived information shall be documented with an access log. These records shall be protected against fire, theft, loss, environmental deterioration, vermin and, in the case of electronic records, electronic or magnetic sources.

5.12.2.5 If an organization goes out of business or changes ownership before the time period for records retention has expired, all documentation shall be transferred in whole to the archives of the sponsor (client) of the work or to the new owner as described in Section 4.1.8.

Legal Issue - See Paul Schuda's language - legal counsel may be required. Refer to language on Annapolis working copy.

5.12.3 Sample Custody Requirements - Essential Documentation

5.12.3.1 Sample Handling - Sample custody shall document all procedures and activities to which a sample is subjected. These activities shall include but are not limited to:

- Sample preservation including appropriate sample container and compliance with holding time;
- Sample identification, receipt, acceptance or rejection and log-in;
- Sample storage and tracking (includes shipping receipts, transmittal forms, and internal routing and assignment records);
- Sample preparation (includes cleanup and separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents, etc.);
- Sample analysis;
- Standard and reagent origin, receipt, preparation, and use;
- Equipment receipt, use, specification, operating conditions and preventative maintenance;
- Calibration criteria, frequency and acceptance criteria;
- Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- Method performance criteria including expected quality control requirements;
- Quality control protocols and assessment;
- Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;

- All automated sample handling systems;
- Records storage and retention; and
- Sample disposal including the date of sample or subsample disposal and name of the responsible person.

5.12.3.2 Laboratory Support Activities - In addition to documenting all the above-mentioned activities, the following shall be retained:

- All original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- Copies of final reports;
- Archived standard operating procedures;
- Correspondence relating to laboratory activities for a specific project;
- All corrective action reports, audits and audit responses;
- Performance evaluation results and raw data; and
- Data review and cross checking.

5.12.3.3 Analytical Records - The essential information to be recorded on all raw data associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, shall include:

- Laboratory sample ID code;
- Date of analysis;
- Instrumentation identification and instrument operating conditions/parameters (or reference to such data);
- Analysis type;
- All calculations (automated and manual); and
- Analyst's or operator's initials/signature.

5.12.3.4 Administrative Records - The following shall be maintained:

- Personnel qualifications, experience and training records

- Initial and continuing demonstration of proficiency for each employee

- A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record

5.12.4 Legal or Evidentiary Custody Procedures

The use of legal chain of custody (COC) protocols is strongly recommended and may be required by some state or federal programs. In addition to the records listed in 5.12.3 and the performance standards outlined in 5.12.1 and 5.12.2, the following protocols shall be incorporated if legal COC is implemented by the organization.

5.12.4.1 Basic Requirements

The chain of custody records shall establish an intact, contiguous record of the physical possession, storage and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates. For ease of discussion, the above-mentioned items shall be referred to as samples:

a) The COC records shall account for all time periods associated with the samples.

b) The COC records shall include signatures of all individuals who had access to individual samples.

c) In order to simplify record-keeping, the number of people who physically handle the sample should be minimized.

d) The COC records are not limited to a single form or document. However, organizations should attempt to limit the number of documents that would be required to establish COC.

e) Legal chain of custody shall begin at the point established by the federal or state oversight program. This may begin at the point that cleaned sample containers are provided by the laboratory or the time sample collection occurs.

f) The COC forms shall remain with the samples during transport or shipment.

g) If samples are shipped, the shipping container shall be sealed in such a manner so that tampering by unauthorized personnel is immediately evident.

h) If required, individual sample containers shall be sealed in such a way to prevent tampering.

5.12.4.2 Required Information in Custody Records

In addition to the information specified in 5.11.1.1 and 5.11.1.2, tracking records shall include, by direct entry or linkage to other records:

a) Time of day and calendar date of each transfer or handling procedure;

b) Signatures of all personnel who physically handle the sample(s);

c) All information necessary to produce unequivocal, accurate records that document the laboratory activities associated with sample receipt, preparation, analysis and reporting; and

d) Common carrier documents.

5.12.4.3 Controlled Access to Samples

Access to all legal samples and subsamples shall be controlled and documented.

5.12.4.4 Transfer of Samples to Another Party

Transfer of samples, subsamples, digestates or extracts to another party are subject to all of the requirements for legal chain of custody.

5.12.4.5 Sample Disposal

a) If the sample is part of litigation, disposal of the physical sample shall occur only with the concurrence of the affected legal authority, sample data user and/or submitter of the sample.

b) All conditions of disposal and all correspondence between all parties concerning the final disposition of the physical sample shall be recorded and retained.

c) Records shall indicate the date of disposal, the nature of disposal (i.e. sample depleted, sample disposed in hazardous waste facility, sample returned to client), and the name of the individual who performed the task.

5.13 LABORATORY REPORT FORMAT AND CONTENTS

The laboratory shall report results, accurately, clearly, unambiguously and objectively and in a manner that is understandable to the recipient. The basic information to be included in the report includes the following:

a) Report title, such as "Certificate of Results" or "Laboratory Results" with the name, address and phone number of the laboratory (or laboratories, see subcontracted laboratories below);

b) Name and address of client and/or project;

c) Description and identification of sample (including client ID code;

d) Date of sample receipt, sample collection and sample analysis;

e) Time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to 48 hours;

f) Test method or unambiguous description of any non-standard method;

g) Test results with any failures or deviations from methods or quality control criteria identified (i.e., data qualifiers);

h) Signature or name, if electronic, and title of individual(s) accepting responsibility for the content of the report and date of issue;

i) Clear identification of any results that were performed by a subcontracted laboratory;

j) Clear identification of all data provided by outside sources, such as air temperature or ambient water temperature;

k) A description of the transformations, calculations, or operations performed on the data, a summary and analysis of the data, and a statement of conclusions drawn from the analysis.

   i. If applicable, identification of whether data is calculated on a dry weight or wet weight basis; and

   ii. In the case of Whole Effluent Toxicity, identification of the statistical package used to provide data.

l) Identification of the reporting units, such as ug/l or mg/kg;

If appropriate, the laboratory shall certify that the test results meet all requirements of NELAP or provide reasons and/or justification if they do not.

Once issued, the laboratory report shall remain unchanged. Any corrections, additions and/or deletions from the original reports shall be supported by supplementary documentation, shall clearly identify its purpose, and shall contain all reporting requirements specified above.

5.14 SUBCONTRACTING ANALYTICAL SAMPLES

5.14.1 If a laboratory subcontracts with another laboratory, the criteria specified in Section 1.6.1.11 shall be followed.

5.14.2 The laboratory shall ensure and have the ability to demonstrate that the subcontracted laboratory meets the criteria and shall advise the client in writing of its intention to subcontract samples.

5.14.3 All records pertaining to the qualifications of subcontracted laboratories shall be maintained, including records of any applicable certifications.

5.15 OUTSIDE SUPPORT SERVICES AND SUPPLIES

The criteria specified in 1.6.1.12 shall be followed.

5.16 COMPLAINTS

The criteria specified in Section 1.6.1.13 shall be followed.

APPENDIX A
REFERENCES

TECHNICAL REFERENCES

ISO/IEC Guide 2: 1986. General terms and their definitions concerning standardization and related activities

International vocabulary of basic and general terms in metrology (VIM): 1984. Issued by BIPM. IEC. ISO. and OIML

ISO 8402: 1986. Quality - Vocabulary

ISO 9000: 1987. Quality management and quality assurance standards - Guidelines for selection and use

ISO 9001: 1987. Quality Systems - Model for quality assurance in design/development, production, installation and servicing

ISO 9002: 1987. Quality systems - Model for quality assurance in production and installation

American Association for Laboratory Accreditation 1993. General Requirements for Accreditation.

ISO 25: 1990. General requirements for the competence of calibration and testing laboratories

QAMS Document *Fred to Provide*

"Guidance on the Evaluation of Safe Drinking Water Act Compliance Monitoring Results from Performance Based Methods", September 30, 1994, Second draft.

METHOD REFERENCES

These methods or method compendiums shall be used when analyzing samples when specified by the accrediting authority or program. The most current version recognized by statute or rule shall be used.

REFERENCES FOR WATER, SEDIMENTS, SOILS, SLUDGES, HAZARDOUS WASTES AND BIOLOGICAL ANALYSES

DRINKING WATER

1) 40 CFR Part 141, National Primary Drinking Water Regulations, Subpart C and Subpart I.

2) "Methods for the Determination of Organic Compounds in Drinking Water," EPA 600/4-88-039, December 1988.

3) "Methods for Chemical Analysis of Water and Wastes," EPA 600/4-79-020, revised March 1983.

4) "Manual for Certification of Laboratories Analyzing Drinking Water, Criteria and Standards Quality Assurance" EPA 570/9-90-008, April 1990 and the first update (Change I) EPA 570/9-90-008a, October 1991.

5) 40 CFR Part 136, Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Appendix A.

6) Standard Methods for the Examination of Water and Wastewater, APHA-AWWA-WPCF.

SURFACE WATER, GROUNDWATER, AND WASTEWATER MUNICIPAL/INDUSTRIAL EFFLUENTS

1) 40 CFR Part 136, Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Tables IA, IB, IC, ID and IE.

2) Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, revised March 1983.

3) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), as amended by updates.

4) 40 CFR Part 261, Identification and Listing of Hazardous Waste, Appendix III (Chemical Analysis Test Methods)

5) Standard Methods for the Examination of Water and Wastewater, APHA-AWWA-WPCF.

Notes:

1) Laboratories analyzing samples in support of NPDES Permits are limited to methods specified in Reference 1 above or those specifically approved for use by EPA.

SOILS AND SEDIMENTS, MUNICIPAL AND INDUSTRIAL SLUDGES (RESIDUALS) AND SOLID AND HAZARDOUS WASTES

1) "Test Methods for Evaluation of Solid Waste, Physical and Chemical Methods", Third Edition (EPA SW-846), as amended by updates.

2) "Procedures for Handling and Chemical Analysis of Sediments and Water Samples" EPA/Corps of Engineers, EPA/CE-81-1.

3) *USEPA Contract Laboratory Statement of Work for Inorganic Analysis".

4) *USEPA Contract Laboratory Program Statement of Work for Organic Analysis".

5) "POTW Sludge Sampling and Analysis Guidance Document" USEPA Permits Division.

* Methods from these references shall be used by laboratories participating in the EPA Contract Laboratory Program to perform analyses for Superfund (CERCLA) site investigations.

AIR

1) Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA 600/4-89/017.

2) 40 CFR Part 50, National Primary and Secondary Ambient Air Quality Standards.

3) 40 CFR Part 53, Ambient Air Monitoring Reference and Equivalent Methods.

4) 40 CFR Part 58, Ambient Air Quality Surveillance.

5) "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II--Ambient Air Specific Methods." EPA-600/ (need to locate latest update)

BIOLOGICAL

Microbiological

1) Drinking Water Analyses - 40 CFR Part 141, Subpart C (Monitoring and Analytical Requirements, section 141.21).

2) Water and Wastewater Analyses - 40 CFR Part 136, Table IA.

3) "Microbiological Methods for Monitoring the Environment" EPA-600/8-78-017, 1978.

4) Standard Methods for the Examination of Water and Wastewater, APHA-AWWA-WPCF.

5) Yearbook of Standards, American Society for Testing Materials. Philadelphia, PA.

6) Official Method of Analysis, AOAC International, Arlington, VA.

7) Manual of Clinical Microbiology, American Society for Microbiology, ASM Publications, Washington, DC.

8) Manual of Industrial Microbiology and Biotechnology, American Society for Microbiology, ASM Publications, Washington, DC.

9) Catalogue of Bacteria, American Type Culture Collection, Rockville, MD.

Bioassay

1) "Methods for Measuring the Acute Toxicity of Effluent and Receiving Waters to Freshwater and Marine Organisms" EPA 600/4-90-027F, August, 1993.

2) "Short-Term Methods for Estimating the Chronic Toxicity of Effluent and Receiving Waters to Freshwater Organisms" EPA 600/4-91-002, September, 1994.

3) "Short-Term Methods for Estimating the Chronic Toxicity of Effluent and Receiving Waters to Marine and Estuarine Organisms" EPA 600/4-91-003, July, 1994.

Macrobenthic Identification and Enumeration

1) "Macroinvertebrate Field and Laboratory Methods for Evaluating the Biological Integrity of Surface Waters", ORD, Washington, D.C., November, 1990.

2) Standard Methods for the Examination of Water and Wastewater, APHA-AWWA-WPCF.

RADIOCHEMISTRY

1) 40 CFR Part 141.25, "Analytical Methods for Radioactivity".

2) Analytical Methods for Radiochemistry Analyses, EPA 600/4-80-032 and EPA 600/5-84-006.

3) Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment", U.S. Nuclear Regulatory Commission Regulatory Guide 4.15.

4) Measurement of Quality Assurance for Radioaassay Laboratories", ANSI N42.2.

APPENDIX B
DEFINITIONS FOR QUALITY SYSTEMS

Accreditation: the process by which an agency or organization evaluates and recognizes a program of study or an institution as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one.

Accrediting Authority: the agency having responsibility and accountability for environmental laboratory accreditation and who grants accreditation. For the purposes of NELAC, this is EPA, other federal agencies, or the state.

Accrediting Body: the organization that actually executes the accreditation process, i.e., receives and reviews accreditation applications, reviews QA documents, reviews performance evaluation testing results, surveys the site, etc., whether EPA, the state, or contracted private party.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Batch: a quantity of material, such as samples, produced or processed in the same operation, and considered to be sufficiently uniform so as to be regarded as a discrete unit.

Batch-lot: the samples having sufficiently uniform attributes (e.g., of like matrix) so they may be processed as a group. *[Florida's definition: Samples which are analyzed (or sampled together) with the same method sequence, the same lots of reagents and with the same treatment common to all samples. The samples must have been analyzed (or collected) within the same specified time period or in continuous sequential time periods. Samples in each set should be a similar composition.*

Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Standard: a solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The Calibration solutions are used to calibrate the instrument response with respect to analyte concentration. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Compromised Samples: those samples which were improperly sampled, or with insufficient documentation (chain of custody and other sample records and/or labels), improper preservation and/or containers were used, or the holding time has been exceeded. Under normal conditions compromised samples are not analyzed. If emergency situations require analysis, the results must be appropriately qualified.

Equipment Blank (Sample Equipment Blank): a clean sample (e.g., distilled water) that is collected in a sample container with the sample-collection device and returned to the laboratory as a sample. Sampling equipment blanks are used to check the cleanliness of sampling devices. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Field Blank: a clean sample (e.g., distilled water), carried to the sampling site, exposed to sampling conditions (e.g., bottle caps removed, preservatives added) and returned to the laboratory and treated as an environmental sample. Field blanks are used to check for analytical artifacts and/or background introduces by sampling and analytical procedures. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Holding Times (Maximum Allowable Holding Times): the maximum times that samples may be held prior to analysis and still be considered valid. (40 CFR Part 136).

Initial Demonstration of Analytical Capability: procedure to establish the ability to generate acceptable accuracy and precision which is included in many of the EPA's analytical methods. In general the procedure includes the addition of a specified concentration of each analyte (using a QC check sample) in each of four separate aliquots of laboratory pure water. These are carried through the entire analytical procedure and the percentage recovery and the standard deviation are determined and compared to specified limits. (40 CFR Part 136).

Instrument Blank: a clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Laboratory: a facility engaged in the collection or analysis and reporting of environmental samples, whether fixed or mobile.

Laboratory Control Sample (quality control sample): an uncontaminated sample matrix spiked with known amounts of analytes from a source independent of the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

Legal Chain of Custody (COC): an unbroken trail of accountability that ensures the physical security of samples, data and records. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Manager (however named): the individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.

Matrix Spike (spiked sample, fortified sample): prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Matrix Spike Duplicate (spiked sample/fortified sample duplicate): a second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Method Blank: a clean sample processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Method Detection Limit (Analytical Detection Limit): the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B).

NELAC: National Environmental Laboratory Accreditation Conference. A voluntary organization of state and federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP.

NELAP: the overall National Environmental Laboratory Accreditation Program of which NELAC is a part.

PBM: Performance Based Methods.

Performance Evaluation Program: the aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results in comparison to peer laboratories and the collective demographics and results summary of all participating laboratories.

Performance Evaluation Sample (PE): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified performance limits. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Preservation: refrigeration and or reagents added at the time of sample collection to maintain the chemical and or biological integrity of the sample.

Pure Reagent Water: shall be ASTM Type I or Type II water in which no target analytes or interferences are detected as required by the analytical method.

Quality Assurance Plan: a formal document describing the management polities, objective, principles, organizational authority, responsibilities, accountability and implementation plan of a laboratory for ensuring quality in its products and utility to its users.

Quality Assurance: an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Quality Control: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Quality Control Sample: an uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Reagent Blank (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Sample Container: a receptacle of suitable material and closure (specified by the program or method) to be used to collect and/or transport samples for testing. The specific requirements for sample containers are to assure a representative samples and sample integrity, e.g., septa vials, glass or plastic.

Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Supervisor (however named): the individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Technical Director: *Definition needs to be developed*

Technical Employee: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent Quality Controls to meet the required level of quality.

Trip Blank: a clean matrix that is carried to the sampling site and transported to the laboratory for analysis without having been exposed to sampling procedures. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

APPENDIX C

PERFORMANCE CHECKLIST

[To be Added Later]